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2009-1002**

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# **SPINEL AS EXIT APERTURE WINDOW FOR HEL SYSTEMS**

**Ishwar Aggarwal et al.**

**Naval Research Laboratory  
Washington DC, 20375**

**17 November 2008**

**Interim Report**

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# Spinel as exit aperture window for HEL systems

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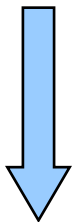
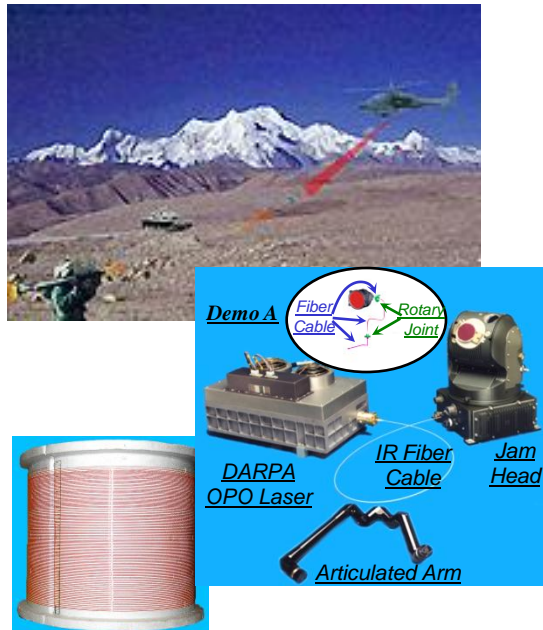
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Presented at the Eleventh Annual Directed Energy Symposium  
November 17-21, 2008  
Honolulu, Hawaii

# Optical Materials – NRL

## IR Fibers for IRCM

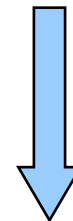


**Rare Earth doped**  
**Mid-IR Fiber Lasers**

## BGG Glass Windows for Recon and HEL

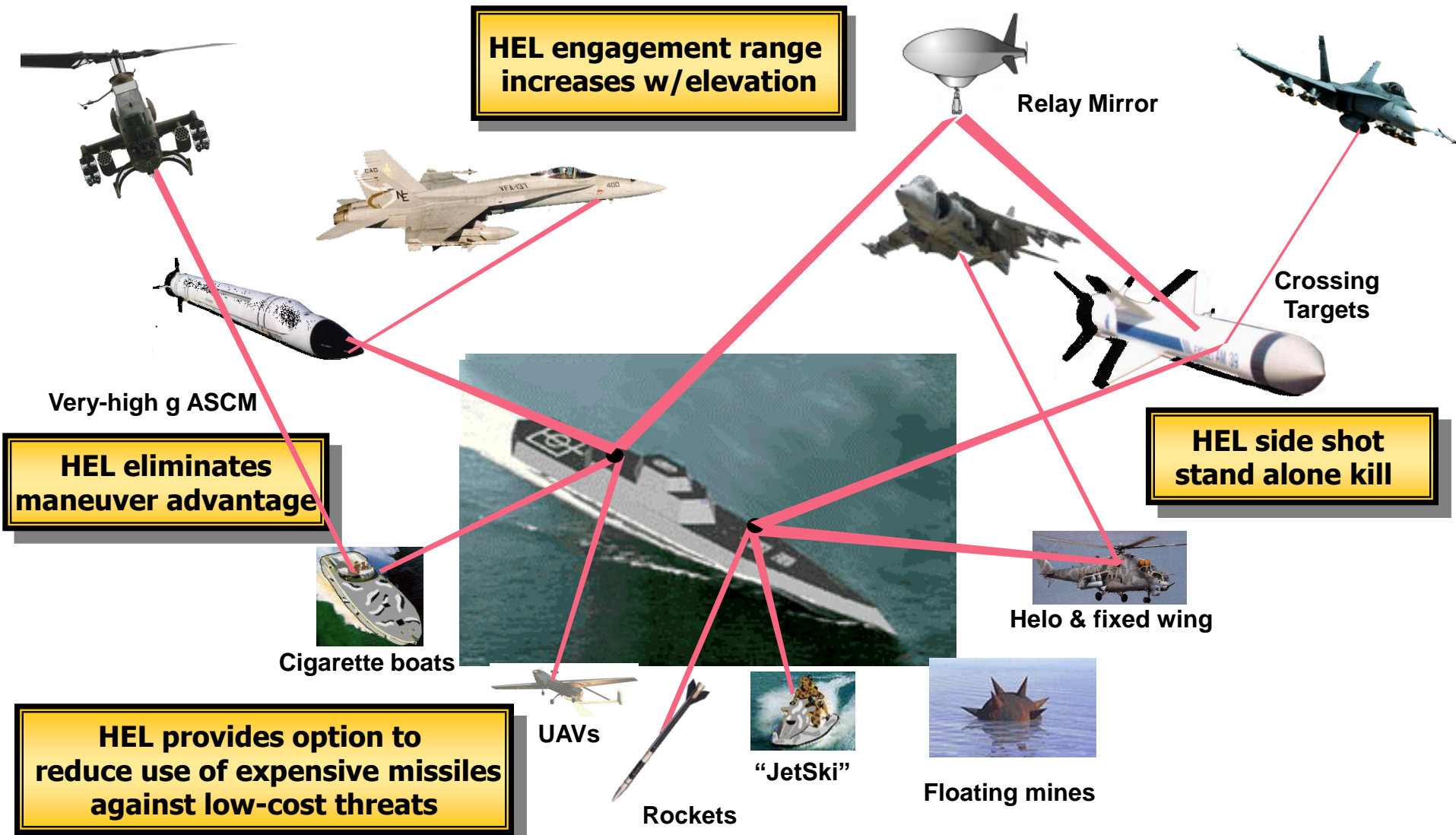


## Spinel Transparent Ceramic Armor



**Rare Earth doped**  
**Solid State Ceramic**  
**Lasers**

# Directed Energy Weapons for Force Protection and Self Defense



# Rugged Windows Needed for Hostile Environments

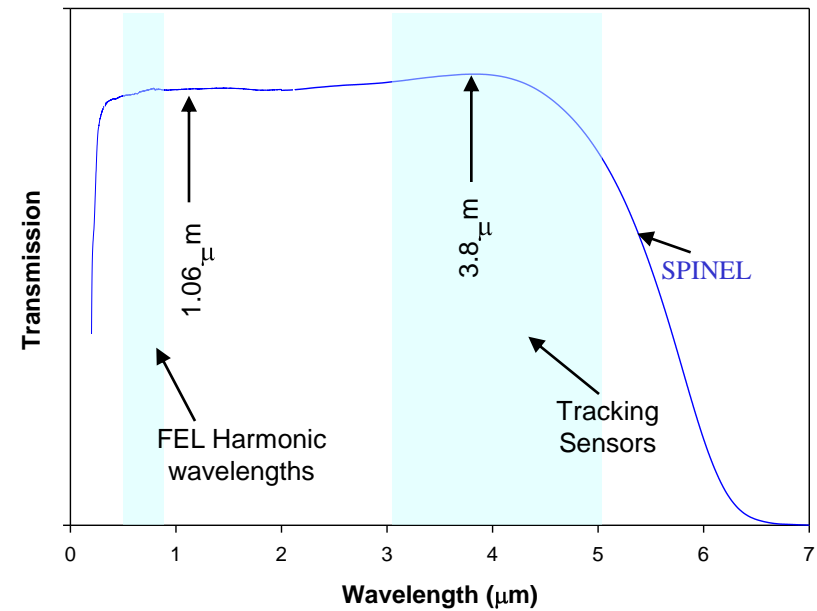


- Current windows materials are limited in hostile environments
  - Low damage tolerance under threat from projectiles
  - Very soft with poor environmental durability
  - Low strength and excessive weight requires extra support
  - High Optical Path Distortion (OPD)

***Single Point of  
Failure for System***

# SPINEL as a Window Material

- NRL has developed breakthrough technology for making transparent SPINEL
  - Excellent transmission 0.2-6.0  $\mu\text{m}$
  - Low absorption loss
  - Excellent UV transmission at FEL harmonic wavelengths
  - Excellent environmental ruggedness
  - Process scalability to large windows
  - Excellent MWIR transmission for tracking and pointing sensors



**Vis-IR transmission of SPINEL**

# Property Comparison with Other Materials

Property Measurements	Fused Silica	OFG Glass	SPINEL
<b><u>Optical</u></b>			
Absorption Coefficient (ppm cm <sup>-1</sup> at 1.06 μm)	12	75	6
Refractive Index (at 1.06 μm)	1.45	1.45	1.707
dn/dT (/K) at 633 nm	1.2x10 <sup>-5</sup>	-9.2x10 <sup>-6</sup>	2.3 x10 <sup>-5</sup>
Stress Optic Coefficient (/Pa)	3.4x10 <sup>-13</sup>	4.1x10 <sup>-13</sup>	3x10 <sup>-13</sup>
<b><u>Mechanical</u></b>			
Density (g/cm <sup>3</sup> )	2.2	3.75	3.58
Poisson's Ratio	0.17	0.31	0.27
Hardness (kg/mm <sup>2</sup> )	600	500 (est)	1645
Fracture Strength (MPa)	50	102	306
Young's Modulus (GPa)	74.5	69.6	271
<b><u>Thermal</u></b>			
Thermal Expansion Coeff. (/K)	0.5x10 <sup>-6</sup>	14.9x10 <sup>-6</sup>	5.9 x 10 <sup>-6</sup>
Heat Capacity C <sub>p</sub> (J/g/K)	0.74	0.67	0.604
Thermal Conductivity (W/(m.K))	1.38	0.7	13.407

## SPINEL compared to Fused Silica:

- 2x lower absorption coefficient
- > 2.5x harder and 6x stronger
- 10x higher thermal conductivity

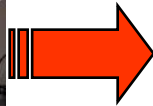
## SPINEL compared to OFG glass:

- >10x lower absorption coefficient
- 3x stronger and > 3x harder
- 3x lower CTE
- 20x higher thermal conductivity

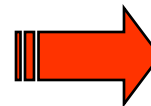
# NRL Process for Making Spinel



*Powder Preparation*



*Hot Pressing*



*Hot Isostatic Pressing*

*Step 1: Prepare high purity Spinel powder*

*Step 2: Hot press powder*

*Step 3: Hot isostatically press to clear transparency*

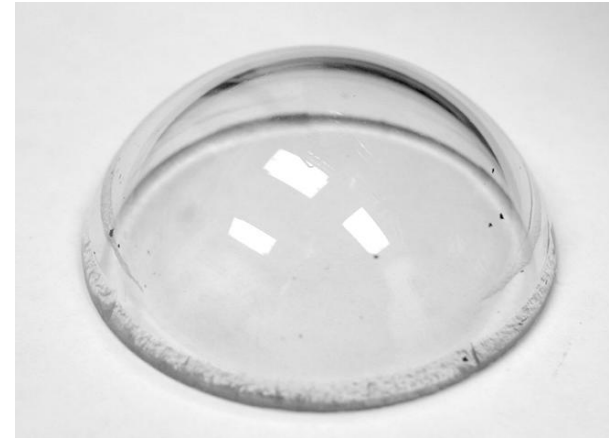
***Simple and inexpensive process***

# NRL SPINEL

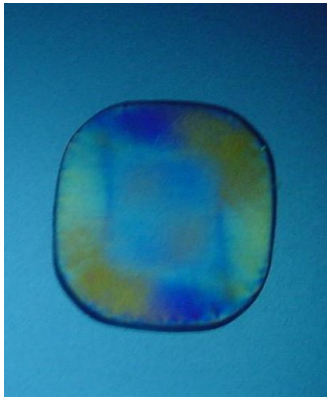
Clarity of spinel



Spinel dome fabrication feasibility



Polarizing Image of the SPINEL Samples

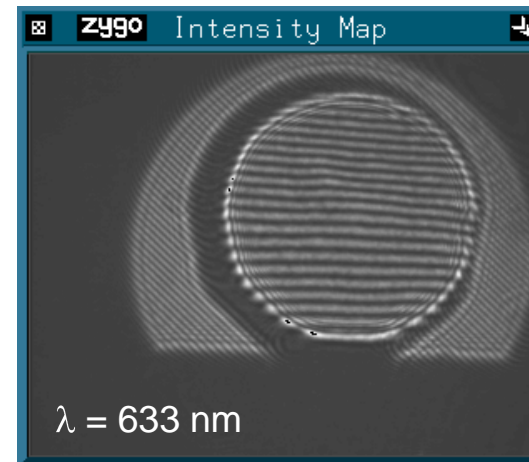


Commercial Single  
Crystal SPINEL  
showing strain



NRL Ceramic SPINEL is  
strain-free

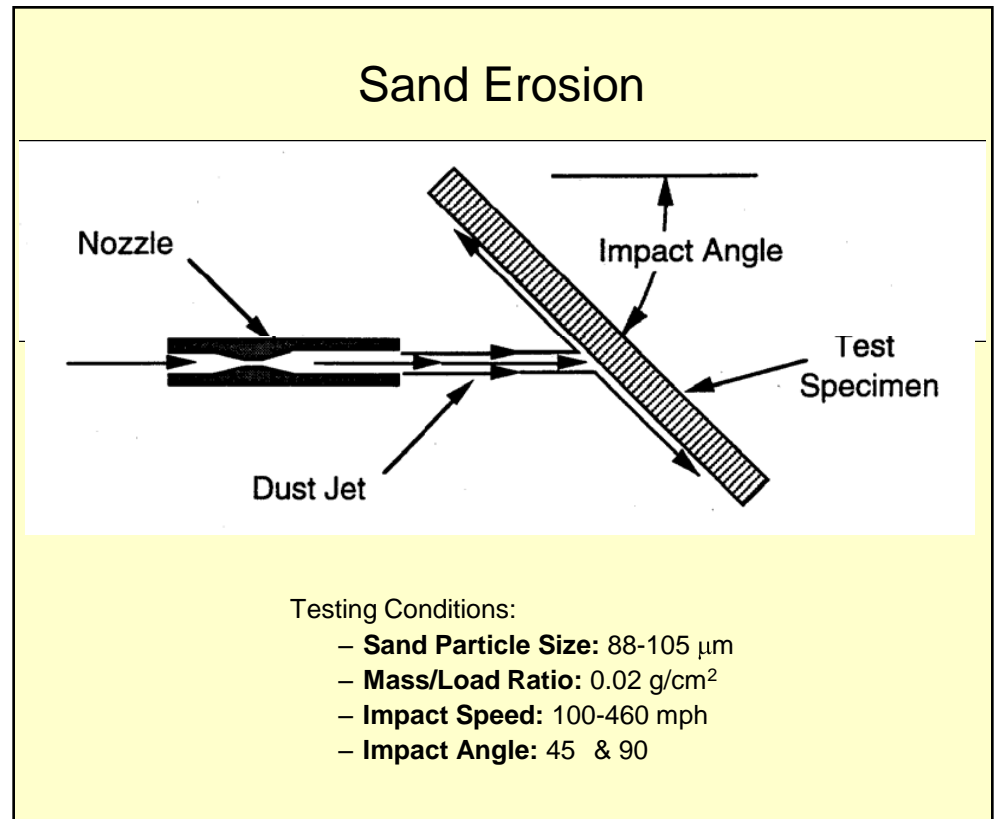
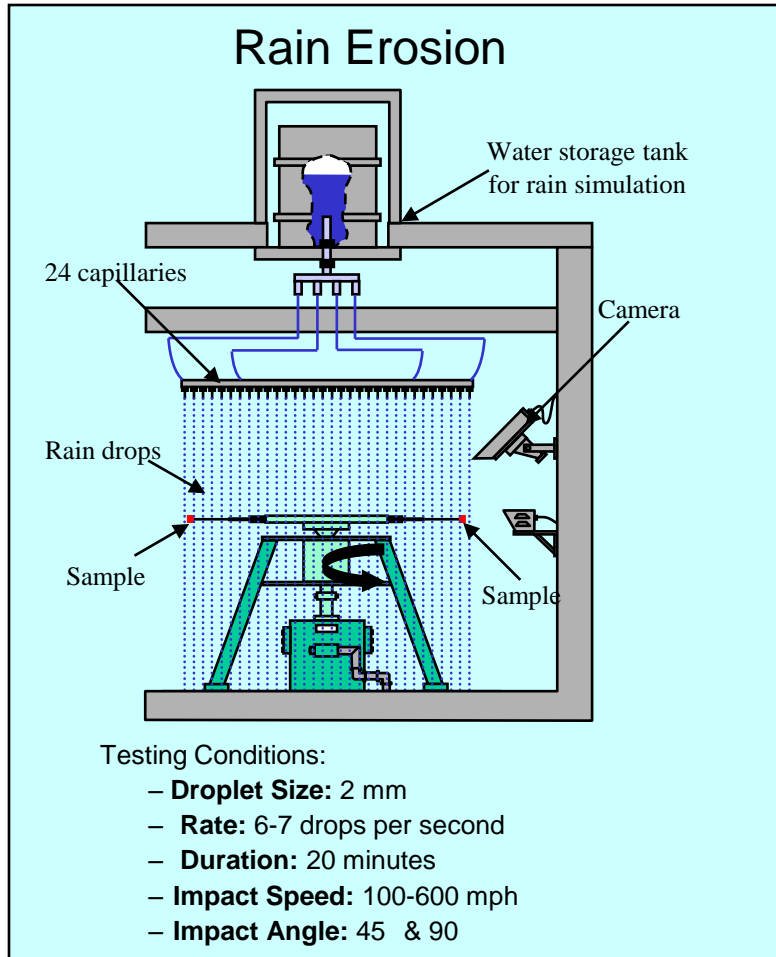
Interferogram



Excellent index homogeneity with transmitted  
wavefront better than  $\lambda/10$  at 633 nm

**NRL SPINEL possesses excellent optical quality**

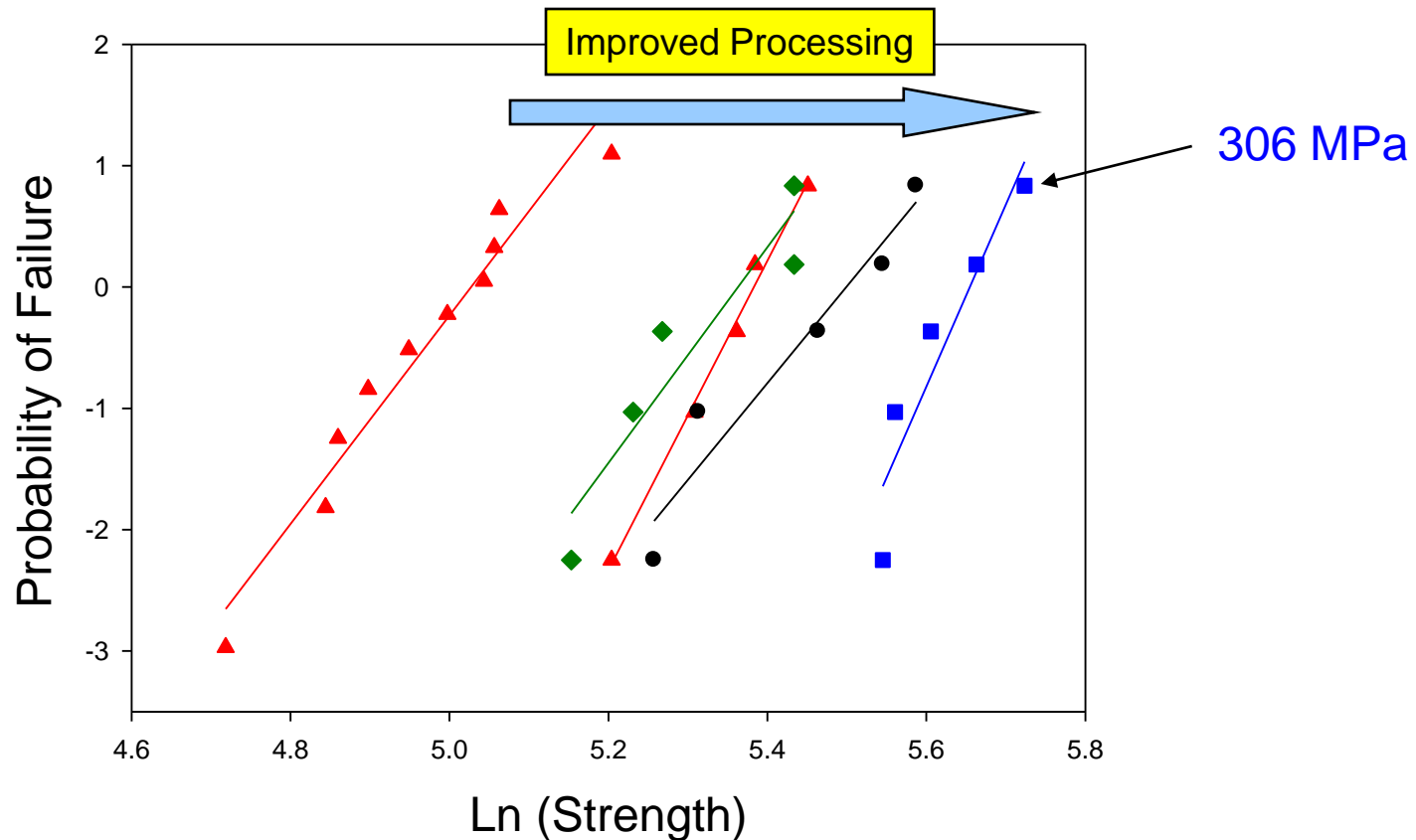
# Rain and Sand Erosion Testing of SPINEL



- Rain and sand erosion testing was performed at Wright Patterson AFB
- SPINEL samples successfully passed the tests

**SPINEL windows can withstand harsh environmental conditions**

# Strength of SPINEL Ceramic

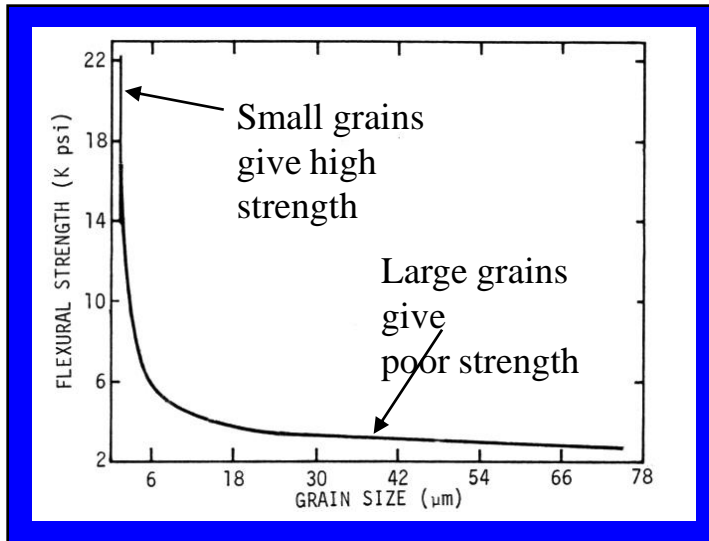


- Increased strength of SPINEL with improved processing control (Not optimized yet)
- Potential to further increase strength of SPINEL
  - Smaller grain size
  - Improved surface quality

# Theoretical Reasoning for Strengthening of SPINEL

- Theoretical strength of SPINEL ~ 20 GPa  
(1/10<sup>th</sup> of elastic modulus, E = 191 GPa)
- Measured strength (150 MPa) is lower due to:
  - Low Fracture Toughness - large grains have poor crack deflection mechanism
    - Reduce grain size and increase fracture toughness
      - Start with nano-sized powder
      - Control grain growth during sintering
  - Porosity at the grain boundary
    - Use NRL developed powder preparation and sintering process
  - Grain pull out and subsurface damage from surface during finishing
    - Improve surface quality by employing better polishing

# Strengthening of SPINEL (Grain Size Factor)



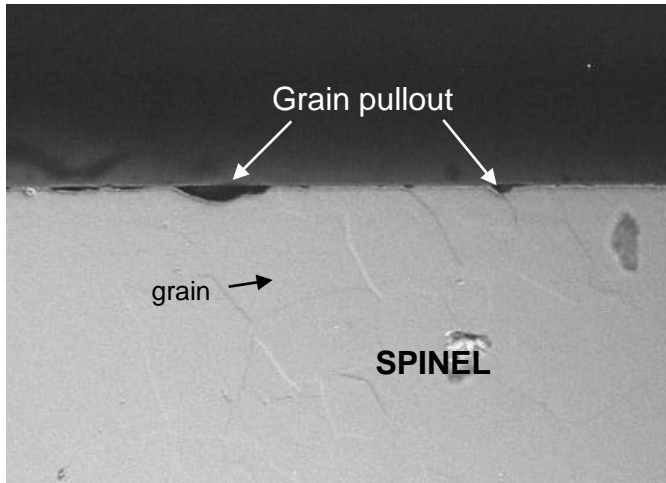
$$\text{Strength} = \sigma_0 + \frac{k}{\sqrt{G}}$$

$\sigma_0$  and  $k$  are constants  
 $G$  = grain size

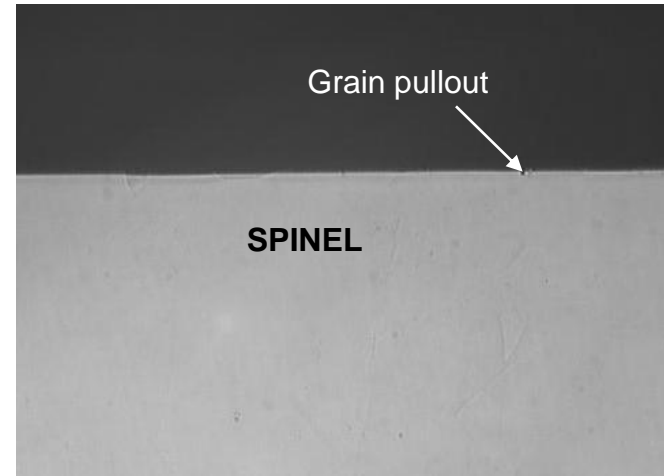
- Demonstrated 306 MPa strength by reducing grain size from 100 μm to 25 μm.
- Grain size will be reduced to ~ 0.5 μm
  - Use SPINEL nanopowder
  - Process improvement to inhibit grain growth
  - Measure strength and fracture toughness

# Strengthening of SPINEL (Surface Finish Factor)

## Improve Surface Finish



Large grain SPINEL. Grain pullout leaves a larger defect. Will have lower strength



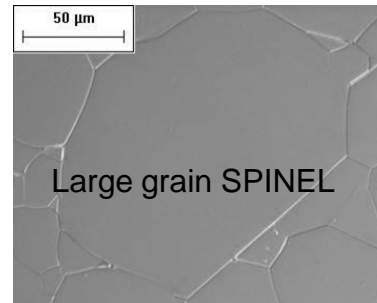
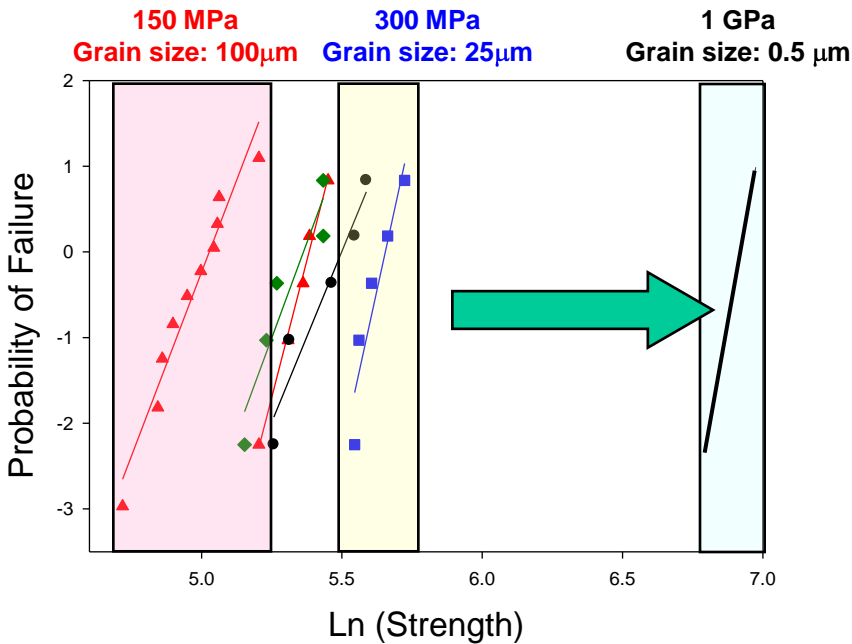
Small grain SPINEL. Grain pullout leaves a smaller defect. Will have higher strength

$$S = \frac{K_{Ic}}{1.24 \sqrt{r}}$$

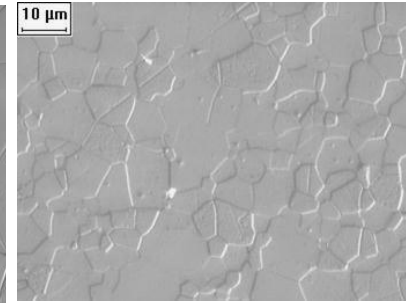
S = Strength  
 $K_{Ic}$  = Fracture Toughness  
r = Flaw size

- Grain pullouts and surface defects create preexisting flaws which lower the strength
- Small grain size will reduce the size of defect from grain pullout → smaller r
- Chemical/mechanical polish (CMP) will be developed to polish SPINEL to better finish
- Two fold improvements have been observed in ceramic strength using CMP

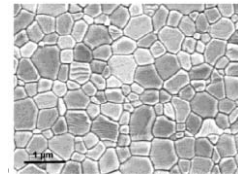
# Strength of SPINEL Ceramic



**Old SPINEL:**  
Grain Size = 100  $\mu\text{m}$   
Strength = 150 MPa



**Current SPINEL:**  
Grain Size = 25  $\mu\text{m}$   
Strength = 300 MPa



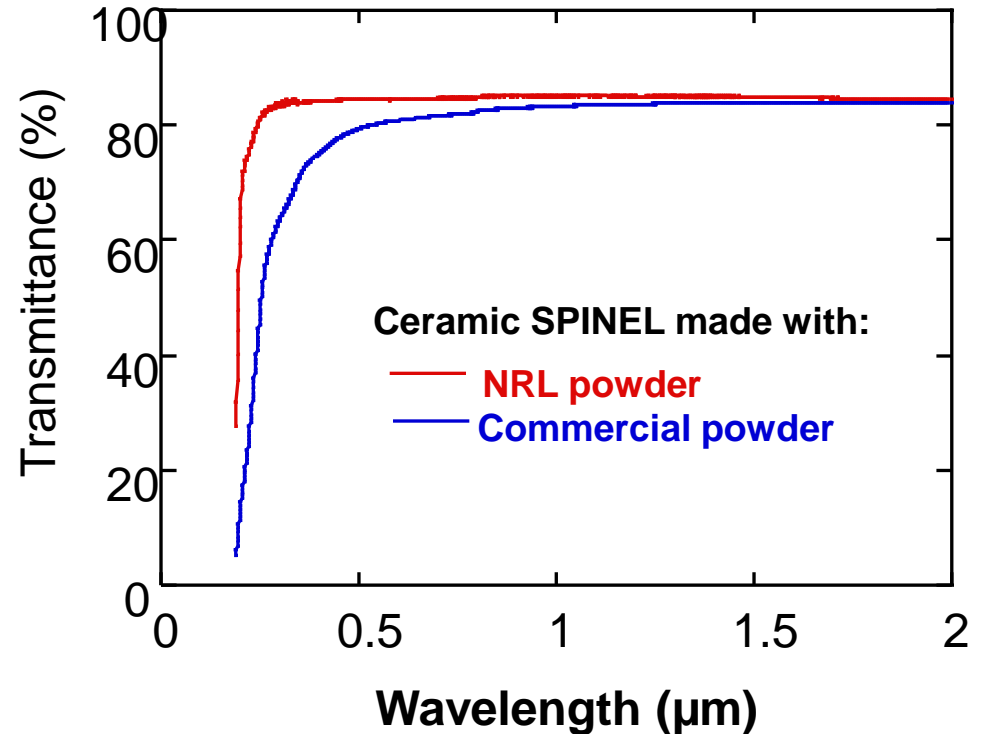
**Future SPINEL:**  
Grain Size = 0.5  $\mu\text{m}$   
Strength >1000 MPa

- Increased strength of SPINEL with improved processing control (Not optimized yet)
- Potential to further increase strength of SPINEL to >1 GPa (>10x stronger than silica)
  - Smaller grain size
  - Improved surface quality

Higher strength will further improve ballistic and HEL performance of SPINEL window

# Purified SPINEL to Reduce Absorption Loss

	Commercial powder (ppm)	NRL powder (ppm)
F	1000	<10
Na	500	4.5
Si	475	18
P	125	15
Cr	175	1.5
Fe	715	4.4
Ni	90	0.40
Y	140	60
Zr	1400	0.24
Ce	365	0.70



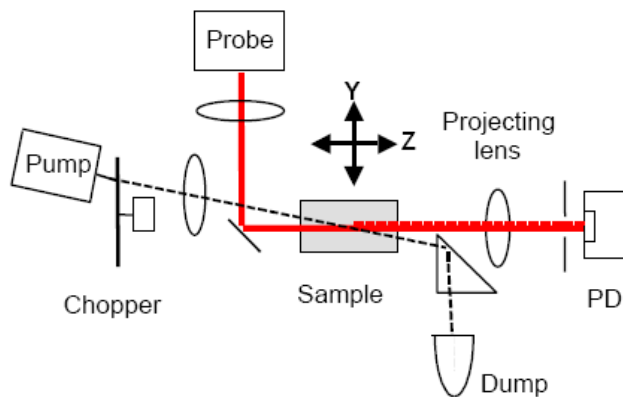
- Fabricated phase pure SPINEL powder using co-precipitation
- Reduced impurity content in SPINEL compared with commercial powder
- Produces reproducible ceramic product
- Ceramic SPINEL made with NRL powder has better UV transmission

# Bulk Absorption Loss in Spinel

## (Measured by PTCPI method at NAWC)

### Photothermal Common-Path Interferometry

- diffraction regime of cross-beam cw thermal lensing -

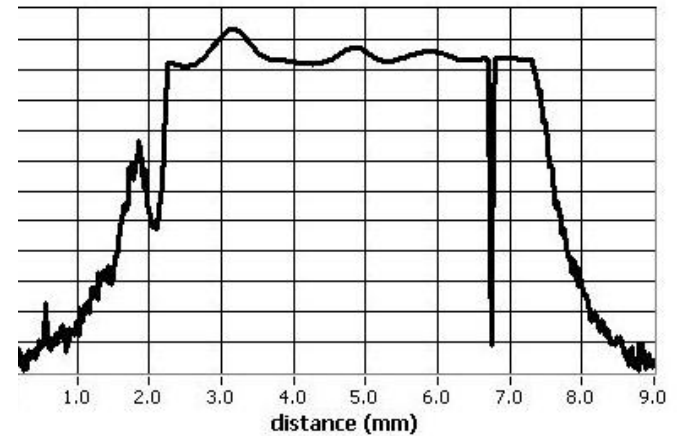


Pump waist	50 $\mu$	Chopping frequency	380 Hz (10Hz- 2 kHz)
Probe waist	120 $\mu$	Crossing angle	1° - 20° (in air)
Pump power	5 W	Probe power	0.5 mW

- ac-component of probe distortion is detected by photodiode + lock-in
- absorption coefficient  $<10^{-7} \text{ cm}^{-1}$  (~10 ppb coating) can be detected with 5 W pump power
- crossed beams help to avoid false signals from optics and surfaces of the sample

Wednesday, October 10, 2007

spinelbulktest5sample2.txt



Bulk Absorption loss in SPINEL

- PTCPI method was used at NAWC to measure bulk absorption in SPINEL samples.
- Bulk absorption loss of 6 ppm/cm was measured at 1064 nm in purified SPINEL samples

# Significance of SPINEL Properties on OPD

## Figure of Merit for OPD ( $FOM_{OPD}$ )

$$FOM_{OPD} = FOM_{\chi} \cdot FOM_{\Delta T} \cdot FOM_L$$

### Thermal distortion FOM ( $\chi$ ):

$$FOM_{\chi} = (1+\nu)(n-1)CTE + \frac{dn}{dT} + n^3 E \cdot CTE (q_{11} + q_{12}) / 4$$

### Temperature rise FOM ( $\Delta T$ ):

$$FOM_{\Delta T} = \frac{\alpha}{\rho \cdot C_P}$$

### Thickness FOM (L):

$$FOM_L = \text{SQRT}((3+\nu) / \sigma_{\max})$$

$\chi$  = Thermal lensing coefficient

L = Window thickness

$\Delta T$  = Window temperature rise

n = index of refraction

$\nu$  = Poisson's ratio

$dn/dT$  = thermo-optic coefficient ( $^{\circ}\text{C}^{-1}$ )

CTE = coefficient of thermal expansion ( $^{\circ}\text{C}^{-1}$ )

$q_{11}, q_{12}$  = stress optic coefficients ( $\text{Pa}^{-1}$ )

E = Young's modulus (Pa)

$\alpha$  = bulk absorption ( $\text{cm}^{-1}$ )

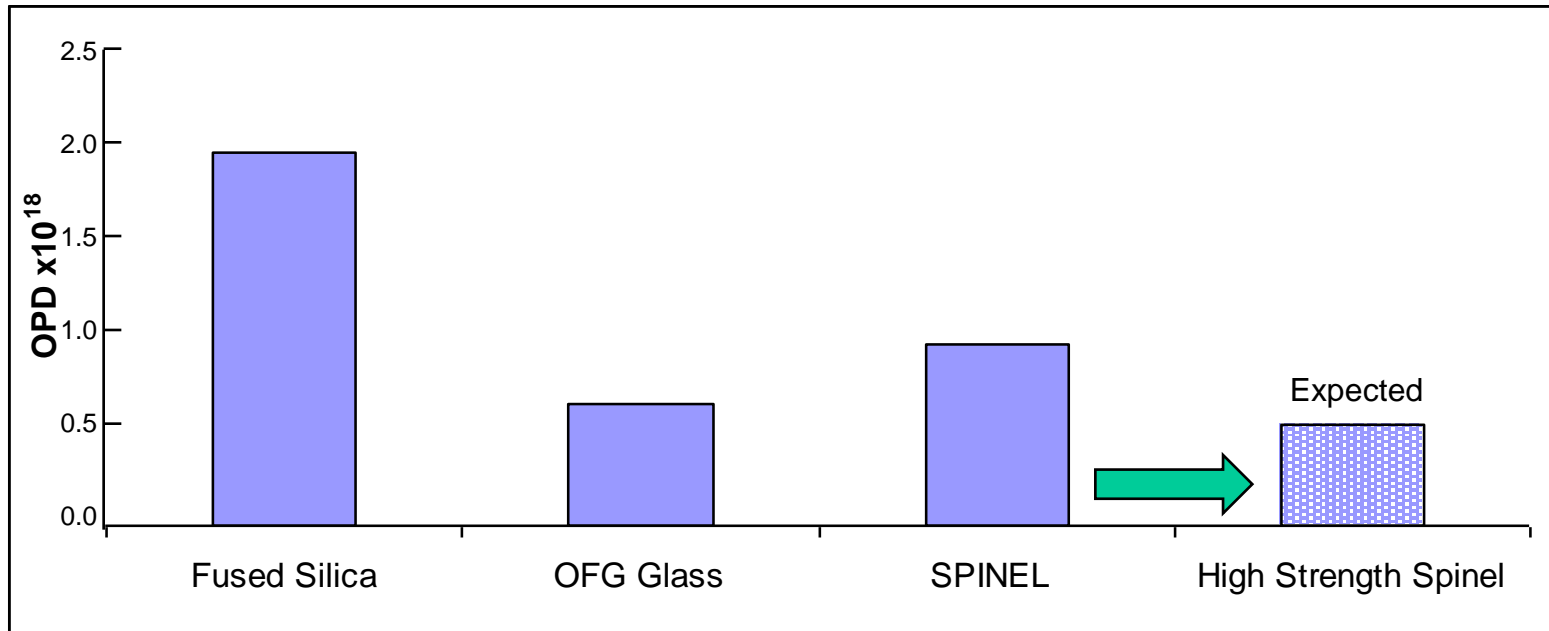
$\rho$  = density ( $\text{gm}/\text{cm}^3$ )

$C_P$  = specific heat ( $\text{J}/\text{g}\cdot\text{K}$ )

$\sigma_{\max}$  = maximum allowable stress (Pa)

- OPD of SPINEL is strongly influenced by  $dn/dT$ , absorption coefficient and strength
- Improvements in SPINEL OPD can be made by:
  - reducing window thickness by improving strength (reduce grain size)

## Figure of Merit for OPD ( $FOM_{OPD}$ )



- OPD of SPINEL is about half of Fused Silica
- Expect OPD of SPINEL to be better than OFG glass with further improvements in strength

# Stress Birefringence

**Stress Birefringence Contribution to Thermal Lensing Coefficient Chi ( $\chi_-$ ):**

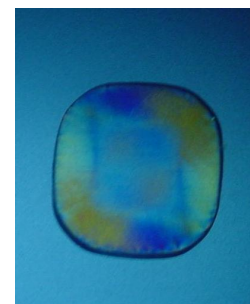
$$\chi_- = (n^3 \cdot E \cdot \text{CTE} / 4) (q_{11} - q_{12})$$

Claude Klein - App. Phys. Letts **87**, 231117 2005

	OFG Glass	SPINEL
n (at 1060 nm)	1.456	1.7
CTE (in /K)	14.60E-06	5.60E-06
Y (in Pa)	6.96E+10	19.35E+10
$q_{11}$ (Pa <sup>-1</sup> )	2.3E-13	3.00E-13
$q_{12}$ (Pa <sup>-1</sup> )	5.9E-13	3.00E-13
$\chi_-$	-2.77E-07	0.00E+00

- OFG glass has large stress-birefringence
- **SPINEL has no stress birefringence due to same  $q_{11}$  and  $q_{12}$**

## Polarizing Image of the SPINEL Samples



Commercial Single  
Crystal SPINEL



NRL Ceramic  
SPINEL

- Single crystal SPINEL exhibits birefringence
- **NRL ceramic SPINEL is strain-free**

**NRL SPINEL will have negligible stress-birefringence contribution to thermal lensing**

# SPINEL Has Good Thermal Shock Resistance

$$\text{Thermal Shock (FOM)} = \frac{S (1 - \nu) k}{\text{CTE} \cdot Y}$$

S = Strength

$\nu$  = Poisson's ratio

k = Thermal conductivity

CTE = Thermal expansion coefficient

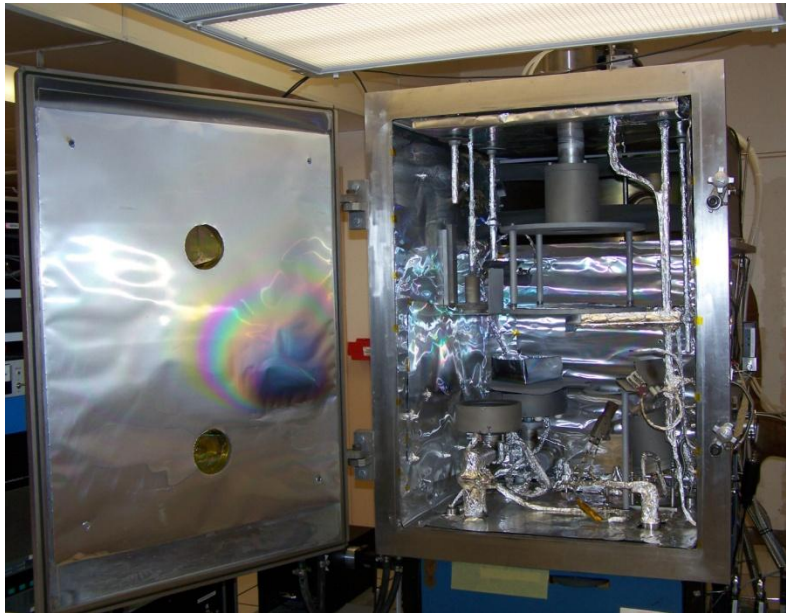
Y = Young's Modulus

	Fused Silica	OFG Glass	SPINEL
CTE (ppm/K)	0.5	14.9	5.9
Strength (MPa)	50	102	300
Youngs Modulus (GPa)	74.5	69.6	282
Poisson's Ratio	0.17	0.31	0.26
Thermal Conductivity (W/m.K)	1.38	0.7	15
<b>Thermal Shock (FOM)</b>	<b>1537</b>	<b>47</b>	<b>2000</b>

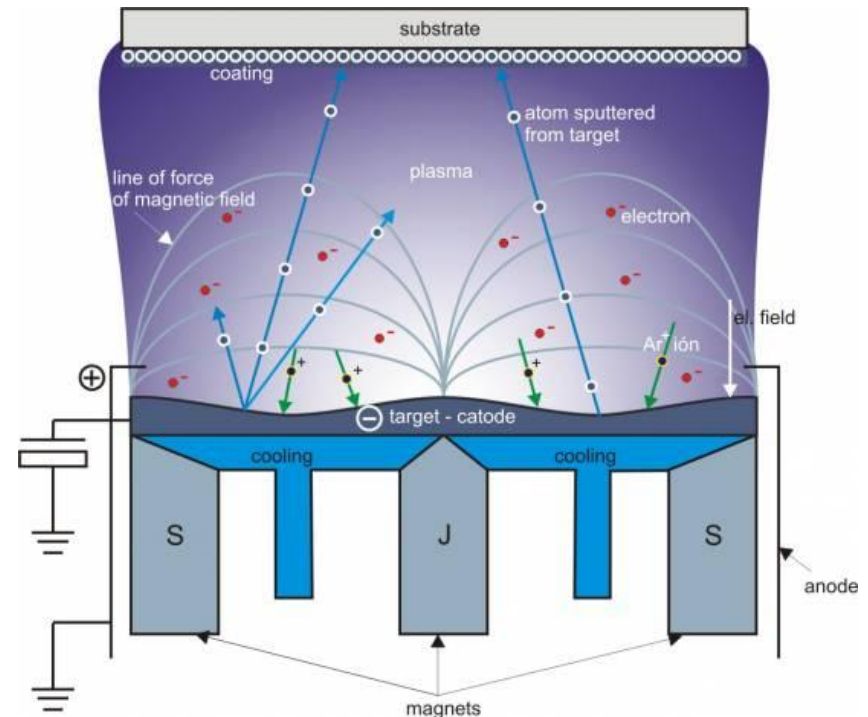
**SPINEL has >40x higher Thermal Shock resistance than OFG glass**

# Application of high damage threshold AR coatings for SPINEL

## DC Magnetron Sputtering



Coating Chamber



- Developed hard abrasion resistant AR coatings using DC Magnetron Sputtering on SPINEL
- Reduced reflection loss from 6.5% to < 0.1% per surface at 1064 nm
- Performed metrology on these coatings for full characterization

# Metrology of AR Coatings on SPINEL

<b><u>Property</u></b>	<b><u>Method</u></b>	<b><u>Measurement</u></b>	<b><u>Results</u></b>
Adhesion	Mil-C-675C	AFRL	Pass
Abrasion	MIL-C-48497	AFRL	Pass
Stress	MiniFlz Interferometer	AFRL	790 MPa (compressive)
Surface Roughness	Atomic Force Microscopy (AFM)	AFRL	$\approx 1$ to 2nm
Absorption Coefficient	Photo-Thermal Common Path Interferometry (PTCPI)	NAWC	65 ppm/cm at 1064 nm

# Adhesion & Abrasion Tests of AR Coatings on SPINEL

## Adhesion Test

- Adhesion tests was done in accordance with MIL-C-675C
  - *The coated optical surface shall show no evidence of coating removal when cellophane tape is pressed firmly against the coated surface and quickly removed at an angle normal to the coated surface.*
- **Passed the Test**

## Severe Abrasion Test

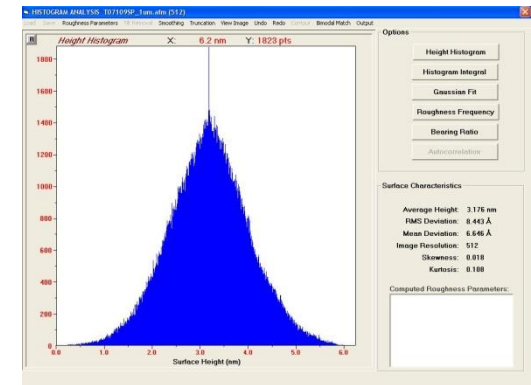
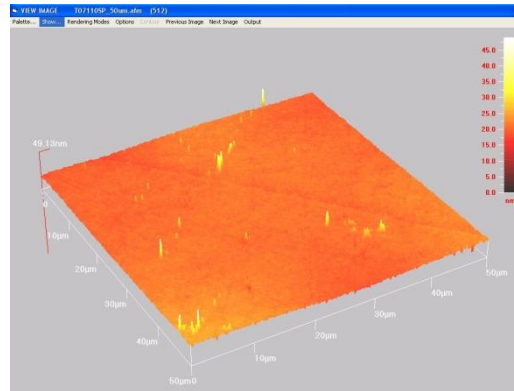
- Severe abrasion test was done in accordance with MIL-C-48497
  - *Abrasion by an eraser conforming to MIL-E-12397 shall not deterioration such as streaks or scratches on the coated optical surface.*
  - *The eraser is made from high grade rubber with 50 5 wt.% abrasive as filler.*
- **Passed the Test**

# Stress and Surface Roughness in AR Coatings on SPINEL

## MiniFiz Interferometer



## Atomic Force Microscopy (AFM)



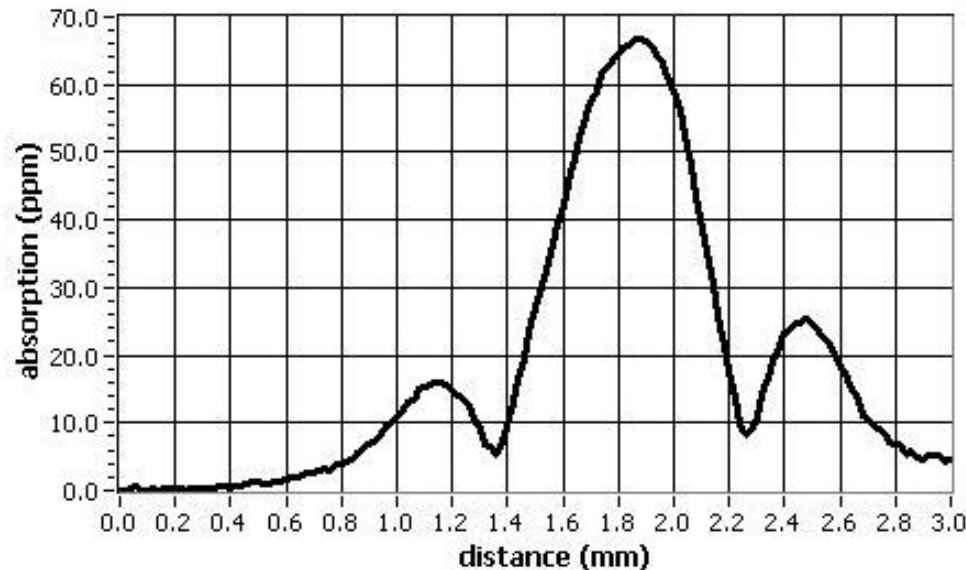
## Stress in coating

- Stress in films was measured using MiniFiz Interferometer
- A 790 MPa compressive stress was measured in AR coatings
- RMS Surface roughness (using AFM) in broad area  $\approx 1$  to 2nm and in smaller local area  $\approx 0.3$ nm Surface roughness primarily substrate surface roughness

# Absorption Loss in AR Coatings on SPINEL

Monday, October 01, 2007

11653b.txt



## Absorption Loss in coating

- Absorption loss in AR coatings measured using Photo-Thermal Common Path Interferometry (PTCPI) method at NAWC
- $\text{ZrO}_2/\text{SiO}_2$  AR coatings demonstrated 65 ppm absorption at 1064nm

# Large SPINEL Window



A 12" x 16" x 1/2" preliminary SPINEL window

- Fabricated a large spinel window
- This demonstrates the feasibility of making large SPINEL windows

# Conclusions

- SPINEL is an Excellent candidate for HEL windows
- Significantly better than current glass materials:
  - > 3x stronger and harder
  - >10x higher thermal conductivity
  - Significantly better thermal shock resistance
  - OPD is comparable to OFG and better than Silica
  - Significantly superior environmental ruggedness
- Successfully developed rugged, low-loss AR coatings for SPINEL
- Demonstrated fabrication of a large SPINEL window

## Pay Off

- Availability of high optical quality AR coated SPINEL windows will have a significant impact on all DoD solid state Directed Energy Weapons Systems operating in harsh environments

## DISTRIBUTION LIST

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